RESEARCH
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CARBON COATED IRON OXIDE FABRICATION AND ITS ELECTROCHEMICAL SENSING APPLICATION

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Abstract

INTRODUCTION: Electrochemical methods of biomolecule sensing is a promising alternative towards conventional methods of estimation, especially in the quantitative analysis of ascorbic acid as they provide a multitude of advantages such as portability, cost effectiveness, delivery of rapid results and non invasiveness.

AIM: To determine the presence and performance of electrochemical sensing ability of carbon coated iron oxide modified electrode towards Ascorbic acid using cyclic voltammetry.

MATERIALS AND METHODS : Solution A consisting of 0.9g of glucose was mixed with 250 ml of water. Solution B consisting of 1.121g of Iron nitrate was mixed with 2 ml of double distilled water. Both solutions were mixed to obtain a third solution which was then subjected to mixing with an ultrasonicator. After which it was transferred to an hydrothermal reactor. The sample was then collected and centrifuged to obtain the final nanoparticle sample, which was subjected to further testing.

RESULTS: Characterization of the nanoparticle sample was conducted using EDX spectrum analysis and XRD analysis from which data confirming the formation of carbon coated iron oxide nanoparticles. The electrochemical sensing capacity of the nanoparticle was analyzed using cyclic voltammetry in comparison to bare carbon electrode. The results show a significant amount of redox activity of ascorbic acid at the corresponding applied voltages.

CONCLUSION: The electrode coated with carbon coated iron oxide nanoparticles were found to have electrochemical sensing activity towards ascorbic acid.

INTRODUCTION

Vitamin c also known as l-ascorbic acid is a water soluble vitamin and a potent antioxidant (1) as well as an essential component required by the human body for its proper functioning. Sources of ascorbate predominantly include fruits and vegetables. One of the most notable human disorders in relation to vitamin C deficiency is scurvy, which results in loss of structural integrity of collagenous structures in the body leading to poor immunity and increased susceptibility to fatal infections like pneumonia(2). However in recent times hypovitaminosis is found to be highly prevalent in the general population, which was observed in various studies(3,4).

Research has been focused on ascorbate on a wide range of industries, most notably in the pharmaceutical industry. It was also found to have various applications in the cosmetic industry and food industry(5)(5,6). The biochemically and physiologically active form of vitamin c consists of L-enantiomer of ascorbic acid with a γ-lactone structure(7). Ascorbate is involved in tyrosine metabolism, folic acid metabolism and cholesterol metabolism(8). Vitamin C is also required for the preservation of collagen because it is required for "hydroxylation" which maintains its structural integrity(9). In order to avoid any pathological condition due to deficiency, a blood plasma level of ≥28 μmol/L of ascorbate should be maintained(3). Regulatory bodies in many countries have tried to recommend the increase of Vitamin C intake to try and counteract hypovitaminosis C(10).

Presently, one of the most common methods for testing vitamin C levels is by Plasma Ascorbic acid test, and leukocyte vitamin c test which involves the collection of blood from the patient and analysis of the isolated plasma(11)(12). These techniques are effective to some extent however they are found to be time consuming and potentially being invasive. Electrochemical sensing methodologies give several advantages over conventional methods such as rapid results, minimal sample requirements, better portability, cost effectiveness and potentially better sensitivity and specificity(13)(14). Significant amount of research is currently undergoing in the field of electrochemical sensing of biomolecules due to their above mentioned advantages(15).

MATERIALS AND METHODS

0.9g of glucose was taken in 40 ml of double distilled water in a 250 ml beaker and was stirred at room temperature. Another solution consisting of 1.121g of Iron Nitrate mixed with 2 ml of double distilled water was taken and added to the glucose solution. Both the solutions were subjected to mixing by an ultrasonicator for 15 minutes. The resulting solution was transferred into a hydrothermal reactor and kept at 190 degrees for 9 hours. After completion of the reaction it was taken out of the hydrothermal reactor and kept for cooling at room temperature. Then the samples were collected and centrifuged many times with water and washed with water and ethanol to remove the unreacted particles. The final nano material was dried in a hot air oven at 80 degree celsius for 12 hours. Finally iron oxide with carbon nanoparticles was obtained.

RESULTS

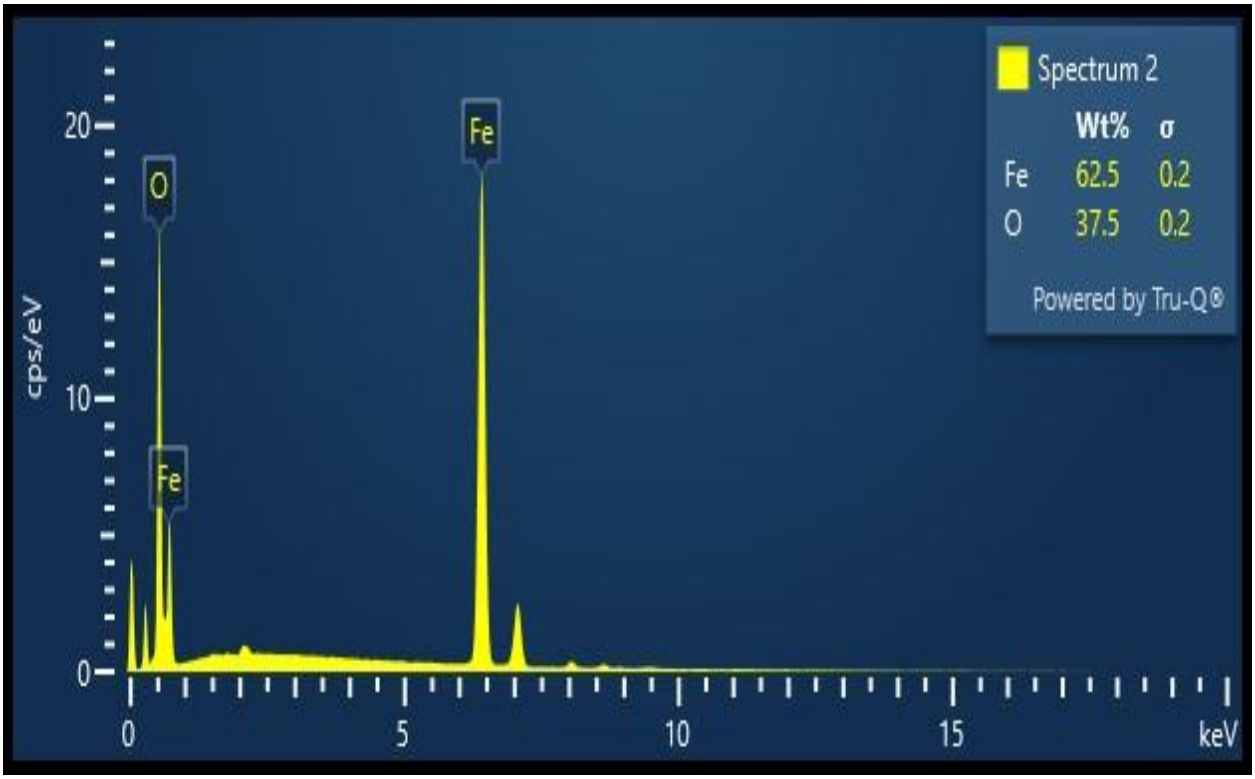

Fig 1:- EDX spectrum analysis of Iron oxide nanoparticle sample.

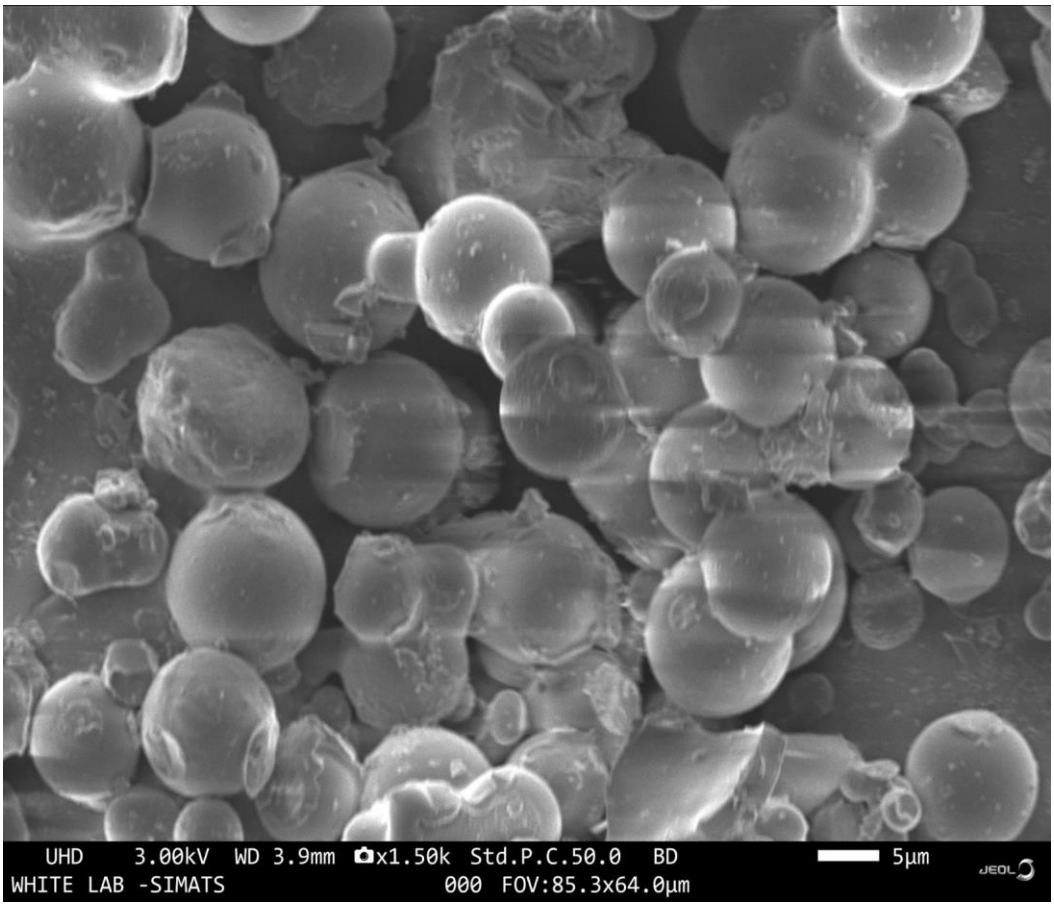

Fig 2:- [a]

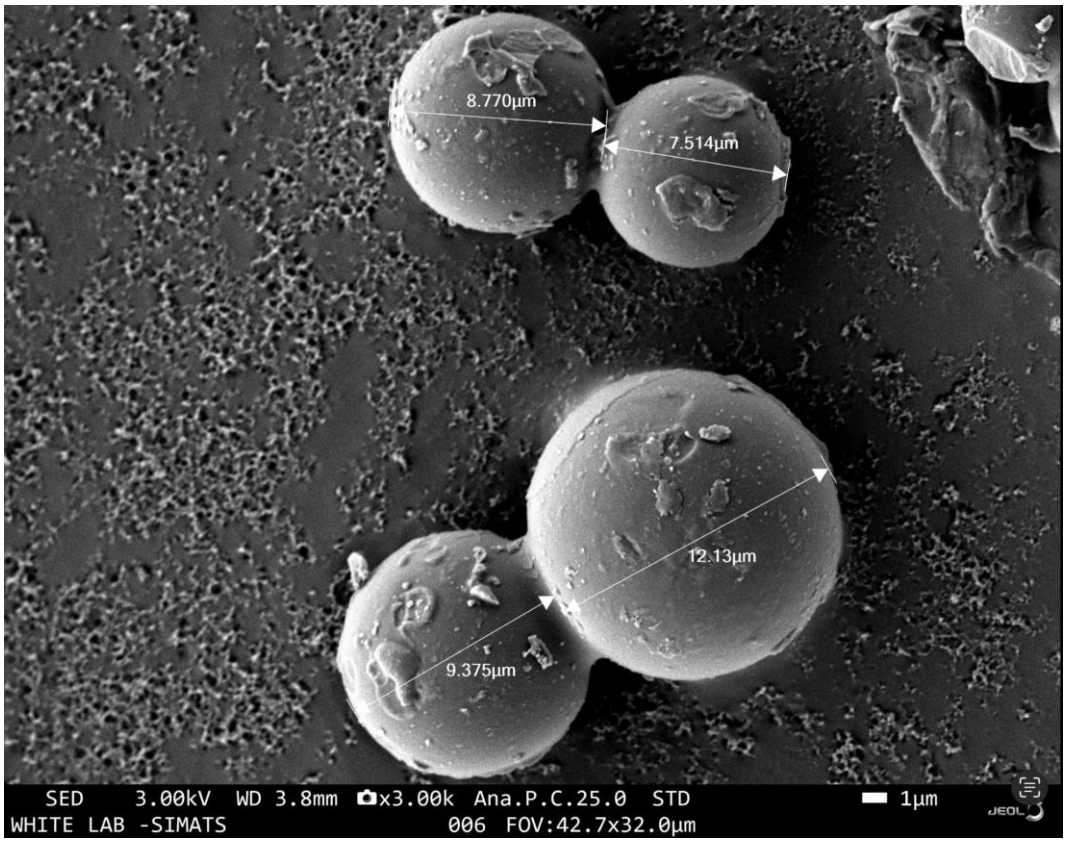

Fig 2:-[b]

Fig 2 :- Field Emission- Scanning Electron Microscopy (FE-SEM) analysis of Iron oxide nanoparticle sample.

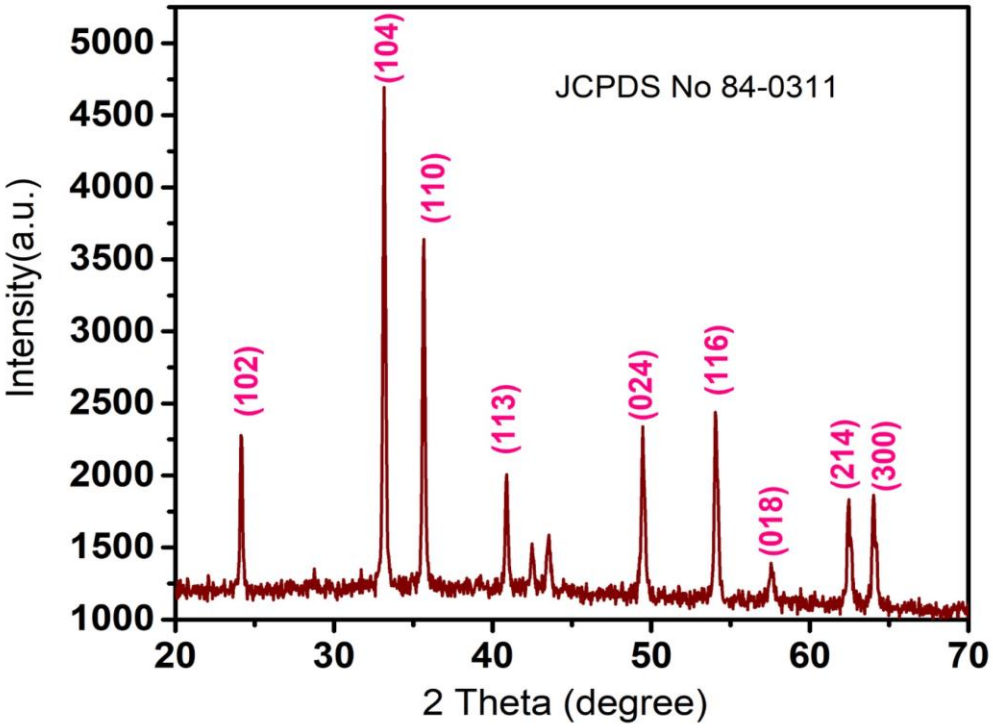

Fig 3:- XRD analysis of the carbon coated Iron Oxide nanoparticles (JCPDS No:- 84-0311)

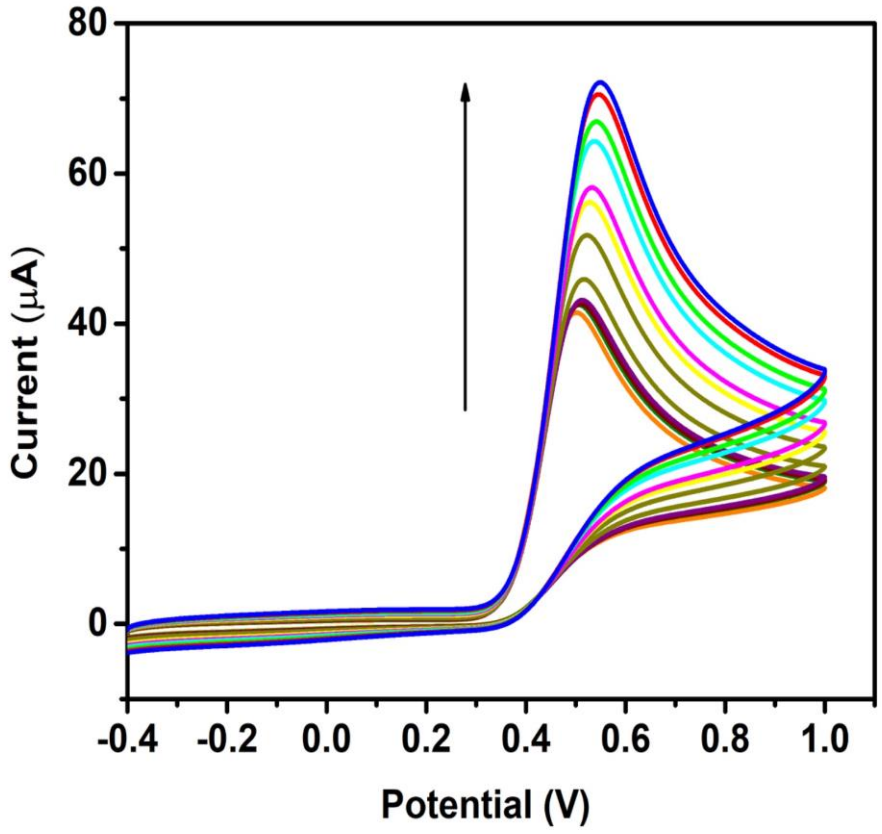

Fig 4:- Effect of Scan rate on cyclic voltammetric response on ascorbic acid at ph 7 with scan rate of 10 to 120 mv/s.

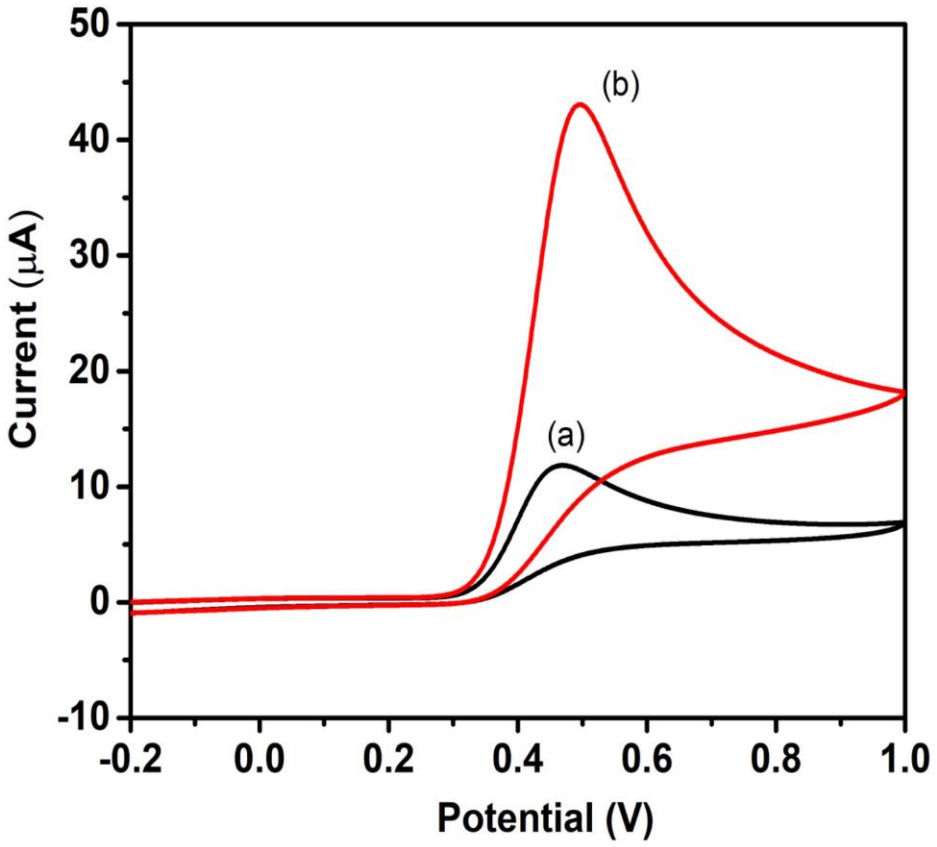

Fig 5:- Cyclic voltammetry response of bare and Carbon Coated Iron Oxide nanoparticles towards Ascorbic Acid in pH of 7 with 50 mv/s

ELEMENTAL ANALYSIS AND CHARACTERISATION:- EDX Spectrum analysis is shown in **Fig 1** indicates the presence of Iron and Oxygen. The XRD spectrum analysis of the nanoparticle sample is shown in **Fig 3**.

SURFACE MORPHOLOGY:- The surface morphology and size of the carbon coated iron oxide nanoparticles were analyzed using Field Emission- Scanning Electron Microscopy (FE-SEM) as shown in **Fig 2**. The particle size was found to be around 8-12 micrometers.

ELECTROCHEMICAL ANALYSIS:- The comparison of cyclic voltammetric response towards ascorbic acid of bare electrode and Carbon coated electrode where a constant voltage of 50 mv/s at an electrolyte pH of 7 applied is shown in **Fig 5,** the black line describes the bare electrode and the red line describes the modified electrode. The effect of scan rate of cyclic voltammetric scan rate towards ascorbic acid at pH 7 with applied voltage ranging from 10 to 120 mv/s at pH 7 is shown in **Fig 4**.

DISCUSSION

From the EDAX Spectrum analysis of the Iron Oxide nanoparticle sample as shown in **Fig 1**, the presence of peaks corresponding to that of iron and oxygen was observed. XRD analysis of the nanoparticle sample was conducted and results were obtained as shown in **Fig 2** was compared with data present in the JCPDS database (JCPDS No- 84-0311) and was found to be a perfect match, hence we can confirm the synthesis of carbon coated iron oxide nanoparticles.

Surface morphology and particle size of the nanoparticle were analyzed using Field Emission Scanning Electron microscopy (FE-SEM) as shown in **Fig 2.** The nanoparticles were found to be spherical in shape with particle diameters ranging from 8 to 12 micrometers. On analysis of the surface morphology of the nanoparticles, carbon particles were found to be coated on the surface.

To check the presence of electrochemical oxidation of ascorbic acid in the modified electrode Cyclic voltammetry was conducted. Cyclic voltammetric responses of bare GCE (a) and Iron oxide/GCE (b) towards ascorbic acid was investigated in phosphate buffer (Na2HPO4+NaH2PO4, pH 7) at the scan rate of 50 mVs-1 **Fig 5**. From the observed cyclic voltammograms it is clear that at GCE, AA is oxidized at 168 mV with the anodic peak current of 11.5 µA and Iron oxide/GCE detects the quercetin with higher current response at 43.05 µA with a lower peak potential of 153 mV. From this observation, it can be seen that Iron oxide/GCE shows enhanced electrochemical sensing property than the bare GCE. The enhanced electrochemical activity is mainly due to the presence of the surface hydroxyl group, smaller crystallite size, and also due to the metal ion. Thus, the Iron oxide/GCE can be utilized for the detection of quercetin at lower potential with enhanced current response.

Effect of scan rate was then conducted to evaluate the performance of the electrode and the results are shown in **Fig 4**. The cyclic voltammetry test was conducted in phosphate buffer (Na2HPO4+NaH2PO4, pH 7) at the scan rate of 50 mVs-1 with applied voltage ranging from 10 to 120 mV/s. Sharp peaks of

current ranging from 40 to 65 micro ampere was observed which shows high levels of Ascorbic Acid redox reaction in the modified electrodes.

From both the cyclic voltammetry studies it is evident that even in very low voltages, the modified was able to produce a signal strong enough to indicate the detection of Ascorbic Acid. The voltage peaks observed in both the cyclic voltammetric tests were found to be very sharp and prominent enough to be considered as a detectable marker.It was also noted that the duration required for the detection of Ascorbic Acid was rapid and specific.

CONCLUSION

The carbon coated Iron Oxide nanoparticles synthesized by Hydrothermal method was tested and utilized for electrochemical and utilized for electrochemical applications if found to have the ability to sense biomolecules. It was confirmed by the electrochemical graphs.

FUTURE SCOPE

Further research can focus on the development of practical sensor devices can be expanded to address specific environmental and biomedical challenges monitoring of pollutants, heavy metals, or biological markers in water, air or biological samples therefore paving the way for innovative applications in various sectors and contributing to the development of sensitive, selective and reliable sensing technologies.

CONFLICT OF INTEREST

There is no conflict of interest.

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AUTHOR CONTRIBUTIONS

All authors are equally contributed.

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